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TPA-active D $-\pi$ -D fluorophores with rigid, planar cores from phenylene to indenofluorene and indolocarbazole

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1. Introduction

Since the pioneering work of Parthenopoulos and Rentzepis [1] and Webb and co-workers [2] on three-dimensional optical data storage and two-photon laser scanning fluorescence microscopy. molecules with strong two-photon absorption (TPA) cross-sections (δ_2) have attracted growing interest [3]. Many chromophores with a large variety of structures have been synthesized and had their TPA cross-sections measured, and fundamental efforts have been made to elucidate structure-property relationships in these systems. Among them, $D-\pi-D$ and $A-\pi-A$ one-dimensional quadrupolar systems have been successfully employed to enhance δ_2 values. The quadrupolar intramolecular charge transfer taking place between either the electron-donating (D) or electron-accepting (A) endgroups and the π -conjugated cores is believed to be an important factor in this enhancement [4]. Further efforts to introduce stronger acceptors or donors as well as elongated cores to enhance this charge transfer have confirmed this assumption [5]. Very large δ_2 values have also been obtained with D-A-D, A-D-A, D-A-D-A-D, and A-D-A-D-A systems [3,6-8]. However, this success is often at the cost of reduced fluorescence quantum yield and/or pronounced redshift of the absorption and emission bands. Blanchard-Desce and

ABSTRACT

A series of $D-\pi-D$ quadrupolar fluorophores with systematically varied π -conjugated cores were prepared using the Heck reaction and their single- and two-photon photophysical properties were examined. Introduction of extended π -conjugated cores such as indenofluorene/indolocarbazole was found to induce only small shifts in the absorption and emission maxima versus the phenylene linked fluorophore but greatly enhance their single- and two-photon absorption cross-sections. The effects of positional substitution on the properties of the indolocarbazole derivatives were also investigated.

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co-workers recently reported a systematic study on the structureproperty relationships of various quadrupolar fluorophores with different types of π -conjugated cores. The results of this work indicated that increasing the connector length between D or A groups induces systematic and pronounced red-shifts and hyperchromic effects on the absorption bands [9].

Most applications of TPA and two-photon excited fluorescence (TPEF) require excitation in the near infrared region (700–900 nm), meaning that a molecular engineering approach to enhance the TPA cross section of the materials without inducing large shifts in their absorption bands would be highly desirable. Although $D-\pi-D$ quadrupolar compound **1** and its fluorene analogue **2** have been exclusively examined as TPA-active compounds [10,11], little attention has been paid to their spectral similarities, particularly the linear absorption and single-photon excited fluorescence emission spectra in dilute solutions, which span nearly the same regions despite their molecular structural differences. Thus, in the current investigation, we have examined the structure-function relationship of $D-\pi-D$ systems with respect to lengthening of the π -conjugated core, in particular focusing on extended fluorene and carbazole-based systems.

These systems, compounds **1–6** in Fig. 1, contain highly rigid and planar π -conjugated cores, with solubilizing alkyl groups. Diphenylamino moieties were chosen as the electron-donating end-groups with styryl bridging units to the varying π -conjugated cores. The linking units employed in this study were: 1,4-phenylene (**1**);





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Fig. 1. Synthesis of quadrupolar fluorophores 1–6. Reagents and conditions: Pd(OAc)₂, P(o-tol)₃, DMF, NEt₃, reflux, 24 h. X = I, for 1, 2 and 4; X = Br, for 3, 5 and 6.

9,9-diethyl-9H-fluoren-2,7-yl (**2**); 9-n-pentyl-9H-carbazole-2,7-yl (**3**); 6,6,12,12-tetra-n-butyl-6,12-dihydroindeno[1,2-b]fluoren-2,8-yl (**4**); 6,12-di-n-octyl-6,12-dihydro-6,12-diazaindeno[1,2-b]fluoren-2,8-yl (**5**) and 6,12-di-n-octyl-6,12-dihydro-6,12-diazaindeno[1,2-b] fluoren-3,9-yl (**6**). Indenofluorene and indolocarbazole moieties have been widely employed in organic light emitting diode (OLED) and organic transistor materials, although there are few reports of their use in TPA chromophores [12, 13]. A compound similar to **4** with methyl rather than n-butyl substituents has recently been described in the patent literature and shown to exhibit high electroluminescent efficiency [14]. Lin and co-workers also reported recently the synthesis and two-photon properties of a multipolar chromophore derived from triphenylamine with three diphenylaminoindenofluorenyl branches [15]. To understand the effects of positional substitution, if any, isomers **5** and **6** were also prepared.

2. Experimental section

2.1. Synthesis and characterization of compounds 1-6

All reactions were carried out under a nitrogen atmosphere using standard Schlenk techniques. NMR spectra were obtained by using Bruker Avance 400 spectrometers. All spectra were recorded in CDCl₃. Chemical shifts are reported relative to tetramethylsilane and are referenced to residual proton or carbon resonances in CDCl₃. Mass spectra were recorded on Applied Biosystems Voyager DE STR (MALDI-TOF) spectrometers. Elemental analyses were performed on a PE 2400 autoanalyser. The melting points were measured on a METTLER-TOLEDO DSC822e differential scanning calorimeter at a heating rate of 20 °C min⁻¹ under nitrogen atmosphere.

General procedure for Heck reaction. A mixture of dihalosubstituted aromatic center (1.0 mmol), 4-N,N-diphenylaminostyrene (0.81 g, 3.0 mmol), tri-o-tolylphosphine (0.15 g, 0.49 mmol), palladium(II) acetate (0.050 g, 0.22 mmol), 10 mL of redistilled triethylamine and 10 mL of redistilled DMF were heated to 80 °C under nitrogen with stirring. After reacting for more than 20 h, the mixture were cooled to room temperature and poured into the water and then extracted with CHCl₃. The organic phase was dried over MgSO₄ and concentrated under reduced pressure and purified by column chromatography on silica gel using chloroform/petroleum ether (1:3 v/v) as the eluent.

3: Yellow powder. Yield 57%. m.p. 212–213 °C; ¹H NMR (CDCl₃, 400 MHz) δ : 0.90 (t, 3H), 1.42 (broad, 4H), 1.90 (m, 2 H), 4.25 (t, 2 H), 7.02–7.46 (m, 36 H), 8.00 (d, J = 8 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ : 12.97, 21.51, 27.74, 28.43, 42.00, 105.55, 116.67, 119.33, 121.35, 121.97, 122.70, 123.45, 126.28, 126.55, 127.24, 128.26, 130.86, 134.40, 140.53, 146.20, 146.59; MS: m/z (%) 676 (M⁺, 100); Anal. calcd for C₅₇H₄₉N₃: C, 88.22; H, 6.36; N, 5.41; found: C, 88.41; H, 6.27; N, 5.37.

4: Yellow powder. Yield 65 %. m.p. 188–190 °C; ¹H NMR (CDCl₃, 400 MHz) δ: 0.59 (m, 20H), 1.02 (m, 8H), 1.97 (m, 8H), 6.95–7.08

(m, 20H), 7.18–7.21 (m, 10H), 7.38 (m, 4H), 7.43 (d, J = 8 Hz, 2H), 7.53 (s, 2H), 7.64 (d, J = 8 Hz, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ : 13.85, 23.10, 26.04, 40.59, 54.60, 113.87, 119.58, 120.61, 122.99, 123.71, 124.50, 125.38, 127.18, 127.26, 127.91, 129.14, 129.28, 131.87, 136.23, 141.07, 147.20, 147.62, 150.45, 151.68; MS: m/z (%): 1016.6 (M⁺, 100); Anal. calcd for C₇₆H₇₆N₂: C, 89.72; H, 7.53; N, 2.75; found: C, 89.76; H, 7.55; N, 2.70.

5: Yellow powder, Yield 52%. m.p. 178–180 °C; ¹H NMR (CDCl₃, 400 MHz) δ : 0.79 (t, J = 8 Hz, 6H), 1.20 (broad, 12H), 1.32 (m, 4H), 1.42 (m, 4H), 1.90 (m, 4H), 4.34 (t, J = 7 Hz, 4H), 6.95–7.07 (m, 14H), 7.14–7.22 (m, 14H), 7.34–7.40 (m, 8H), 7.87 (s, 2H), 8.07 (d, J = 8 Hz, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ : 14.07, 22.63, 27.43, 28.87, 29.25, 29.48, 31.88, 43.30, 98.53, 106.28, 116.73, 120.27, 122.44, 122.78, 122.97, 123.77, 124.46, 127.29, 127.41, 128.49, 129.28, 132.00, 135.46, 136.84, 142.17, 147.117, 147.64; MS: m/z (%) 1018.7 (M⁺, 100); Anal. calcd for C₇₄H₇₄N₄: C, 87.19; H, 7.32; N, 5.50; found: C, 87.31; H, 7.28; N, 5.48.

6: Pale yellow powder. Yield 65%. m.p. 170–172 °C; ¹H NMR (CDCl₃, 400 MHz) δ : 0.86 (t, J = 6 Hz, 6H), 1.26 (broad, 12H), 1.38 (m, 4H), 1.46(m, 4H), 1.96 (m, 4H), 4.41 (t, J = 7 Hz, 4H), 7.01–7.15 (m, 14H), 7.23–7.29 (m, 14H), 7.37 (d, J = 7 Hz, 2H), 7.45 (d, J = 7 Hz, 4H), 7.66 (d, J = 7 Hz, 2H) 8.02 (s, 2H), 8.32 (s, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ : 14.06, 22.63, 27.45, 28.93, 29.23, 29.47, 31.86, 43.53, 99.08, 108.60, 118.23, 122.85, 122.99, 123.29, 124.09, 124.36, 124.57, 125.18, 127.00, 128.14, 128.44, 129.27, 132.70, 136.57, 141.45, 146.77, 147.79; MS: m/z (%) 1018.7 (M⁺, 100); Anal. calcd for C₇₄H₇₄N₄: C, 87.19; H, 7.32; N, 5.50; found: C, 87.26; H, 7.26; N, 5.53.

2.2. Photophysical properties measurement

Linear absorption spectra with $C = 1.0 \times 10^{-6} \text{ mol L}^{-1}$ were recorded on a Shimadzu UV 2550 spectrometer. Single-photon excited fluorescence (SPEF) spectra with $C = 1.0 \times 10^{-6} \text{ mol L}^{-1}$ were measured on an Edinburgh FLS920 fluorescence spectrometer. The SPEF quantum yields Φ for the compound were determined relative to coumarin 307 [16] using a standard method [17]. TPEF experiments with $C = 1.0 \times 10^{-4} \text{ mol L}^{-1}$ were performed with a femtosecond Ti: sapphire laser (80 MHz, 200 fs pulse width, Spectra Physics) as pump source and a USB 2000 + Micro Fiber Spectrometer (Ocean optics) as recorder. More details on the experimental setup and the method can be found in previous work [18]. The TPA crosssections of **1**–**6** in toluene and THF solutions were measured by using TPEF measurement technique and coumarin 307 as reference solution [19].

3. Results and discussion

3.1. Synthesis

As shown in Fig. 1, compounds **1–6** were synthesized via crosscoupling of the appropriate dihalo-substituted arene with two equivalents of 4-N,N-diphenylaminostyrene under typical Heck reaction conditions, catalyzed by Pd(OAc)₂ and P(o-tol)₃, 1,4-diiodobenzene is commercially available. Indenofluorene was prepared starting from 1,4-dibromo-2,5-dimethylbenzene by a similar method to that reported by Wang and co-workers [20] with a little revision. Normally, the direct halogenation of fluorene will take place at 2,7 positions [21]. Similar halogenations with I₂-HIO₃ in acid condition also occur for indenofluorene at para-positions (C2 and C8 positions) in our work. However, the direct halogenations of carbazole always occur at the 3,6 positions. Therefore, the method of Müllen and co-workers was used to prepare the 2,7-dibromocarbazole starting from commercially available 4,4'dibromobiphenyl [22]. The 2,8-dibromo-indolo[1,2-b]-carbazole and 3,9-dibromo-indolo[1,2-b]-carbazole was synthesized by the method reported by Beng and co-workers, with bromine atom being introduced since the starting materials of the first step (4-bromophenylhydrazine hydrochloride and 3-bromophenylhydrazine hydrochloride, respectively) to avoid the difficulty of seperation of isomers in the normal direct halogenations [23a]. To improve the solubility of these compounds, alkyls groups of varying length were introduced at the bridging C or N atoms. Details of the syntheses and characterizations of compounds 3-6 are given in the experimental section, and those for their dihalo-arene precursors are provided in the supporting information.

3.2. Linear absorption and single-photon excited fluorescence

Fluorophores **1–6** all display solubility in common solvents of intermediate polarity, such as toluene, THF and chloroform. Single-photon absorption and fluorescence spectra recorded for **1–6** are shown in Fig. 2, and the relevant photophysical data are summarized in Table 1. To our surprise, they exhibit quite similar photophysical properties despite their structural differences. Examination of the absorption spectra of the related fluorene-based compounds, **2** and **4**, shows that upon extension of the core of these chromophores, a negligible red-shift in their absorption maxima, $\lambda_{max}(abs)$, of less than 2 nm takes place. As shown in Fig. 2a, these compounds display distinct, broad absorption bands with maxima of 411–416 nm, and also longer-wavelength shoulders. It should also be noted that an increase in the length of the conjugated cores results in enhancement of the extinction coefficients (ε). For example, the value of ε for compound **4** is nearly twice that of **1**.

The carbazole-based compounds **3**, **5** and **6** show quite different trends. Although the absorption spectrum of **3** is nearly identical to that of **1**, that of **5** displays a λ_{max} value of 416 nm with a secondary peak at 440 nm, both of which are significantly red-shifted relative to that of **3** (5 nm for λ_{max} and 20 nm for the longer-wavelength shoulders). In addition, changing the position of the electron-donating substituents on the cores substantially alters the spectral properties. Compared with spectrum of **5**, the main absorption band of **6** is blue-shifted by 27 nm, and the value of ε is also decreased by nearly 20%.

In addition to the similarity of the absorption spectra, the fluorescence emission properties of these compounds are also quite similar despite the differences in their molecular structures. As shown in Fig. 2b, compounds **1–4** emit light in the blue-green spectral region with emission maxima, λ_{max} (em), of 450–455 nm. Compound **5** exhibits a maximum at 473 nm, a red-shift of ca. 20 nm. Although linear absorption of **6** is greatly blue-shifted relative to those of others, its emission maximum is similar to those of compounds **1–4** (around 453 nm). All of their emission spectra contain secondary peaks at longer wavelengths. Their fluorescence quantum yields, Φ , are also sizeable (0.60–0.84).

The spectral similarity of these compounds is interesting. Considering the molecular symmetry of compounds 1-5, maybe



Fig. 2. The normalized linear absorption (a) and single-photon excited fluorescence emission spectra (b) of compounds 1-6 in toluene (1.0 μ M).

we can assume that the intramolecular charge transfer during the excitation are also symmetrical, which means the charge transfer only involving nearly the same domains from the electrondonating triphenylamino to the phenyl of central core. What should be pointed out, although the fluorene and indenofluorene keep the bridge with high rigidity and planarity, the phenyls in these cores seems not conjugated very well to each other. For example, we had previously reported crystal structure of indenofluorene [24]. The five-membered rings in indenofluorene do not fit the Hückel aromaticity rule. The bridging C–C bond lengths in indenofluorene are both 1.468 Å, which is significantly longer than those of the phenyl rings (1.391 and 1.404 Å). These less conjugation may also contribute to the spectral similarity.

Table 1	
The photophysical properties of the compounds 1–6 in toluene. ^a	

	$\lambda_{max}(abs)/nm^b$	$\log \varepsilon$	$\lambda_{max}(em)/nm^{c}$	Φ	$\delta_2/\mathrm{GM^d}$
1	412	4.96	455	0.82	696
2	412	5.13	451	0.84	870
3	411	5.10	451	0.81	910
4	413	5.21	450	0.66	1280
5	416	5.36	473	0.62	1770
6	389	5.10	453	0.60	880

^a All single-photon-physical properties were measured as 1.0 µM solutions.

^b Only the longest-wavelength absorption peaks are shown.

^c Emission measurements excited at the absorption maximum.

 d TPA cross-sections were measured by the comparative TPEF method [19] on $1.0\times10^{-4}\,M$ solutions excited by a fs laser at 730 nm with Coumarin 307 as reference.



Fig. 3. Crystal structures of indenofluorene [24] (left) and indolocarbazole [23c] (right). (All the H atoms and most of the C atoms of the solubilizing alkyl groups were omitted for clarity.)

However, the differences between those of compounds **4** and **5** could be related to the differences in the bond lengths of their cores. Differing from that of indenofluorene, the five-membered rings in indolocarbazole fit the Hückel aromaticity rule. As mentioned above, for indenofluorene, the bridging C–C bond lengths are significantly longer those of the phenyl rings. However, in indolo[3,2-b]carbazole [23c], the bridging C–C bonds lengths are 1.443 Å (see Fig. 3), which is closer to those of the analogous phenyls (1.410 and 1.417 Å).

The differences in the optical properties of compounds **5** and **6** could be related to the differences in the positions of their electrondonating groups relative to the indolocarbazole cores. Similar spectral differences were also observed in other indolocarbazole derivatives reported by Leclerc and co-workers [13,23c]. In compound **5**, the electron-donating groups are located at the 2- and 8-positions of the core, so electron-delocalization from terminal amines to the center will be permitted. However, in compound **6**, they are at the 3- and 9-positions, para- to the bridging N atoms, so electron-delocalization from terminal amines to the center will be forbidden, but electron-delocalization from terminal amine to bridging N atoms should be permitted, meaning that the conjugation length of **6** is shorter than that of **5**.

3.3. Two-photon absorption and two-photon excited fluorescence emission

Up to now, the accurate measurement of TPA cross-section value is still difficult to do. The TPA cross section value had been found to depend on the experimental condition, such as the laser, concentration, solvents and the references for TPEF method [3a,3b]. So, even the same compound, the data reported by different group maybe also



Fig. 4. Two-photon excited fluorescence emission spectra of 1--6 in toluene (1.0 \times 10 $^{-4}$ mol L $^{-1}$).

different in many cases. For example, a compound similar to our compound **2** had also been studied with the TPEF technique by Fitilis et al. [11]. However, the TPA value reported in that paper is smaller than both other researcher's [10] and ours. So the comparison between the results from different group should be very careful.

However, the result under the same condition should be reliable to investigate the structure-dependence. In this work, the TPA crosssections of these compounds were measured by the comparative TPEF measurement technique, using femtosecond laser pulses as the light source, as described previously. As with most of the reported quadrupolar TPA fluorophores at low concentration, in all cases the TPEF spectra are quite similar to the single-photon fluorescence spectra, except of lower intensity and with more noise (see Fig. 4). The pronounced vibronic structures in the single-photon data are not as apparent in the two-photon data. This maybe due to the spectrometer slits in the two-photon experiment are normally opened wider to get enough signal level. However, the two-photon excitation spectra contain maxima which are significantly blueshifted with respect to those of single-photon absorption (see Fig. 5). This is likely a consequence of the effect of molecular symmetry as manifested in the parity-derived selection rules [3]. It well known, that the spectral selection rule of TPA $(g \rightarrow g)$ is different from that of single-photon absorption $(g \rightarrow u)$ for the centrosymmetric structures. But both SPEF and TPEF emission processes, with only one photon involved, should abide by the same rule $(u \rightarrow g)$.

Being limited by the wavelength range of our laser system, we are unlikely to have found the maximum TPA cross-sections. However, based on the δ_2 values obtained at 730 nm, as given in Table 1, it seems that elongation of the central core significantly enhances the TPA. The TPA cross-section of compound **5** is as large as 1770 GM at 730 nm, which is comparable to those of other reported efficient TPA systems. However, the TPA cross-section of compound **6** is only 880



Fig. 5. Two-photon excitation spectra of **1–6** in toluene $(1.0 \times 10^{-4} \text{ mol L}^{-1})$.

GM at 730 nm, probably related to its shorter conjugation length. This result is consistent with the prediction that symmetric charge transfer is correlated to enhance values of δ_2 .

4. Conclusion

In conclusion, we have synthesized a series of $D-\pi-D$ systems with different cores, ranging from benzene to indenofluorene and indolocarbazole. Systematic comparison of their single- and two photophysical properties reveals that the systems with longer cores, such as indenofluorene and indolocarbazole have significantly enhanced one- and two-photon absorptions, but only small shifts in their absorption or emission bands.

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